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Low temperature redetermination of the structure of $[Ni(CO)(\eta^5\text{-}C_5H_5)]_2$

Cameron Evans* and Louis J. Farrugia

Department of Chemistry, University Of Glasgow, Joseph Black Building, University Avenue, Glasgow G12 8QQ, Scotland

Correspondence e-mail: cevans@chem.gla.ac.uk

Key indicators

Single-crystal X-ray study
T = 115 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.024
wR factor = 0.065
Data-to-parameter ratio = 20.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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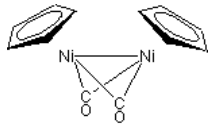
The crystal structure of $[Ni(CO)(\eta^5\text{-}C_5H_5)]_2$ has been redetermined at 115 K. The low temperature data provide a more precise structure solution and indicate the presence of a minor degree of disorder in the material. There are two molecules in the asymmetric unit.

Comment

The structure of $[Ni(CO)(\eta^5\text{-}C_5H_5)]_2$, (I), has been determined previously on two separate occasions, both at room temperature (Byers & Dahl, 1980; Madach *et al.*, 1980). The advances made in crystallographic techniques since this time allowed for a redetermination of the structure at lower temperature with a significant increase in the quality of the structure solution.



(I)

As previously reported, there are two independent molecules in the asymmetric unit (Fig. 1). The Ni—Ni distances [2.3691 (3) and 2.3575 (3) Å for Ni1—Ni2 and Ni3—Ni4 respectively] are greater than those previously reported [2.3627 (9) and 2.3510 (9) Å (Byers & Dahl, 1980); 2.361 (2) and 2.348 (2) Å (Madach *et al.*, 1980)], though the remaining bond parameters of the molecules display no significant differences. Both molecules possess non-planar $Ni_2(CO)_2$ units, analogous to that found for $Co_2(CO)_8$ (Sumner *et al.*, 1964; Leung & Coppens, 1983), with the η^5-C_5H_5 rings tilted in the opposite direction. The angle between the η^5-C_5H_5 and NiC_2 planes ranges from 80.33 (6) to 82.56 (7)° across the two molecules.

A minor component of disorder was noted from difference maps and the positions of the Ni atoms in this component identified. Refinement indicated the extent of the disorder to be approximately 1%, with the Ni—Ni distances in the minor component [2.32 (2) and 2.34 (2) Å for Ni1*a*—Ni2*a* and Ni3*a*—Ni4*a* respectively] similar to those in the main structure. The positions of the Ni atoms in the minor component of the disorder are related to those of the major component by a translation of 6.3–6.4 Å along the *c* axis of the unit cell (Fig. 2). The possibility that this electron density is a result of twinning rather than disorder was examined using the *ROTAX* program (Cooper *et al.*, 2002) to test for the presence of a twofold axis, though none was identified. Similarly, the set comprising the weakest 655 reflections provided a mean scale

factor K (K = mean F_o^2/mean F_c^2) of 1.87, lower than generally observed for a genuine twinned structure. There was no evidence for twinning from the diffraction pattern.

Experimental

$[Ni(CO)(\eta^5\text{-}C_5H_5)]_2$ was obtained from a commercial source (Sigma–Aldrich Ltd). Crystals suitable for structural determination were obtained from a concentrated hexane solution at 253 K. Contrary to previous reports, all crystals obtained through sublimation displayed evidence of twinning and were disregarded.

Crystal data

$C_{12}H_{10}Ni_2O_2$	$Z = 4$
$M_r = 303.62$	$D_x = 1.871$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.7498$ (1) Å	Cell parameters from 133537 reflections
$b = 10.8752$ (1) Å	$\theta = 1$–$35.0°$
$c = 13.4942$ (2) Å	$\mu = 3.47$ mm^{-1}
$\alpha = 76.776$ (1)°	$T = 115$ (2) K
$\beta = 81.112$ (1)°	Prism, black
$\gamma = 78.449$ (1)°	0.40 × 0.28 × 0.15 mm
$V = 1077.65$ (2) Å^3	

Data collection

Nonius KappaCCD diffractometer	5839 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.071$
Absorption correction: multi-scan (Blessing, 1995)	$\theta_{max} = 30°$
$T_{min} = 0.264$, $T_{max} = 0.594$	$h = -10 \rightarrow 10$
84232 measured reflections	$k = -15 \rightarrow 15$
6276 independent reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0315P)^2 + 0.5418P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.025$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.065$	$\Delta\rho_{max} = 0.62$ e Å^{-3}
$S = 1.10$	$\Delta\rho_{min} = -0.64$ e Å^{-3}
6276 reflections	Extinction correction: *SHELXL*
307 parameters	Extinction coefficient: 0.0030 (4)
H-atom parameters constrained	

All H atoms were placed in calculated positions ($U_{iso} = 1.2\ U_{eq}$ of the C atom to which they were attached) using a riding model. The Ni atoms of the minor component of the disorder were identified from difference maps and refined using isotropic displacement parameters.

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

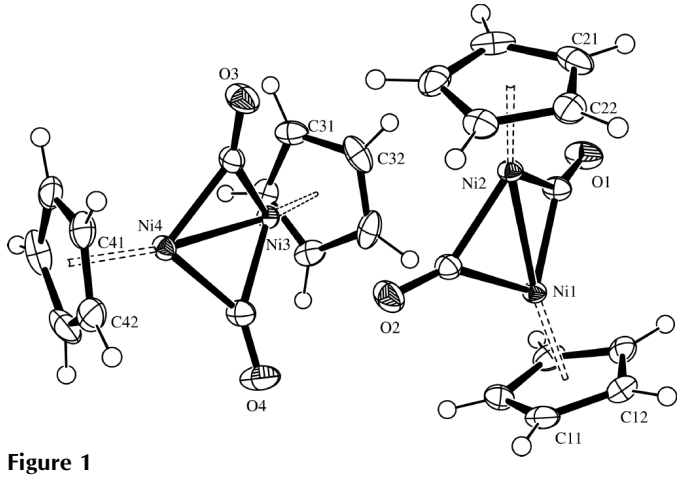


Figure 1
View of the two molecules in the asymmetric unit, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level, with H atoms represented by circles of arbitrary size. The minor component of the disorder has been omitted

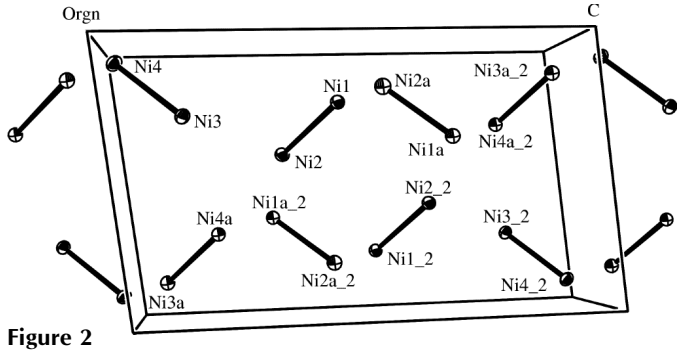


Figure 2
View of the unit cell, indicating the positions of the Ni atoms in both components of the disorder.

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References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Byers, L. R. & Dahl, L. F. (1980). *Inorg. Chem.* **19**, 680–692.
Cooper, R. I., Gould, R. O., Parsons, S. & Watkin, D. J. (2002). *J. Appl. Cryst.* **35**, 168–174.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837.
Leung, P. C. & Coppens, P. (1983). *Acta Cryst.* B**39**, 535.
Madach, T., Fischer, K. & Vahrenkamp, H. (1980). *Chem. Ber.* **113**, 3235–3244.
Nonius (1997–2000). *COLLECT*. Nonius BV, Delft, Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, edited by C. W. Carter Jr and R. M. Sweet, Part A, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Sumner, G. G., Klug, H. P. & Alexander, L. E. (1964). *Acta Cryst.* **17**, 732.